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Note

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Critical behaviour of the thermodynamic quantities near the melting point in the solid phase of hexadecane

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Abstract : We study here critical behaviour of the thermodynamic quantities such as thermal expansivity α_p, isothermal compressibility κ_T and the specific heat C_p close to the melting point in solid hexadecane By analyzing experimental data for the thermal expansivity measured at various pressures for constant temperatures of 302 and 325 K, we calculate the pressure dependence of the isothermal compressibility κ_T and specific heat C_p near the melting point in solid hexadecane We then establish the Pippard relations (C_p *vs* α_p and α_p *vs* κ_T) for this molecular organic compound close to its melting point

Values of the slope dP_m/dT which we deduce from our linear plots of the Pippard relations, are in good agreement with the experimentally measured value for solid hexadecane near the melting point

Keywords : Pippard relations, melting point, solid hexadecane

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1. Introduction

Solid hexadecane as a molecular organic compound (MOC), is one of those long chain paraffin solids and exhibits critical behaviour near the melting point. As pointed out in a previous study [1], it has been shown experimentally that its specific heat C_p at atmospheric pressure exhibits a pronounced premelting in a temperature range of 60 K from the melting temperature [2,3]. This premelting has been determined experimentally as 1.43 cal for *n*-hexadecane between the temperatures of 15.885 and 19.288°C [4]. In the solid and liquid phases of *n*-hexadecane, the specific heat C_p was measured at the triple temperature of T_t^* = 18.175°C and the values were found as 93.30 and 121.11 cal/°C.mole, respectively [4]. This premelting has also been observed experimentally, where the thermodynamic quantities behave abnormally [3,5,6]. Those premelting anomalies have been observed in a variety of physical probes such as

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some low-frequency optical librational modes [7–9], neutron Bragg intensities [10–12], scattered light [13] and the dielectric constant [14], as also indicated by Pruzan *et al* [1].

Recently, experimental studies on hexadecane have been reported in the literature. Some of those studies are the following : The solid-liquid equilibrium (SLE) diagrams have been obtained experimentally for the binary systems of benzene, cyclohexane + *n*-tetradecane, *n*-hexadecane at temperatures 230–323 K and pressures up to 120 MPa [15]. Very recently, the crystallization temperature of two binary mixtures, the tetradecane + hexadecane and tetradecane + pentadecane systems, has been measured experimentally up to 100 MPa [16] and the experimental results have been compared with the predictions of the model developed on solid-liquid equilibria [17]. Also, the melting temperatures of the three compounds (tetradecane, pentadecane and hexadecane) at various pressures have been obtained experimentally [16] with the results of binary systems (tetradecane + hexadecane and tetradecane+ pentadecane) and they have been compared with the literature data [18]. T-X data for a binary mixture of tetradecane + hexadecane [16] have been compared with the data obtained by using the differential scanning calorimetry (DSC) [19] and also using the kinetic [20] and dilatometric [21] methods. The surface tension of hexadecane solutions of F(12)H(16) as functions of temperature and bulk concentration have been measured, and phase transitions of the adsorbed film of $F(CF_2)_{12}(CH_2)_{16}H$ at the surface of liquid hexadecane, have been studied experimentally [22]. It has been indicated in an earlier study [23] that the surface phase transition for hexadecane occurs in the supercooled state. Regarding the phase change materials (PCMs), their thermophysical properties such as the melting and freezing point, the heat of fusion and the specific heat [24], have been studied experimentally. Among the experimental techniques, DSC in particular has been used to investigate the thermal characteristics of the PCMs [25–29]. The other experimental techniques have also been used to study the thermal performance of the storage systems of the phase change materials [30–34]. The phase change materials consist of mostly binary or ternary mixtures of paraffin waxes whose thermal characteristics have been studied experimentally during freezing and melting processes [35]. Experiments have been carried out using a mixture of tetradecane (40%) and hexadecane (60%) with the melting temperature of 6.8°C. Also, the liquid-solid phase equilibrium of binary mixture system of tetradecane and hexadecane has been studied for cool storage [36,37] and their phase transition temperature ranges have been determined experimentally [36] by the DSC. Apart from those studies, interface solvent effects have been investigated experimentally in colloidal phase transitions for *n*-hexadecane, benzene and toluene [39]. In *n*-hexadecane the gel information has been observed experimentally in an earlier work [40].

It has been pointed out that C_p varies less rapidly than the thermal expansivity α_p [1]. On approach to the critical point, C_p is expected to behave like the isothermal

compressibility κ_T and α_p [41]. Using the measurements of the thermal expansivity for the solid hexadecane, the value of 1/2 for the critical exponent of the thermal expansivity was obtained from the analysis according to a power-law formula [1]. Considering the premelting effects in solid hexadecane, this critical behavior indicates that a second order phase transition occurs in the solid phase of this molecular organic compound. In the precursor zone, the specific heat C_p increases with a critical exponent about 0.1 [1]. This increase is much steeper for κ_T and α_p, whereas close to the pseudocritical line, C_v and κ_s remain finite [41]. Since C_p, κ_T and α_p diverge near the melting point, they can be related to each other, as C_p was related to α_p by Buckingham and Fairbank [41]. These thermodynamic relations are usually derived to describe λ-type of phase transitions [42]. Thus, the divergence behaviour of the thermodynamic quantities considered above, close to the melting point in the solid phase of hexadecane has to be investigated extensively. The mechanism of a second order phase transition prior to melting in the solid phase of hexadecane can then be explained by analyzing the thermodynamic quantities critically. From the point of view of phase transitions in hexadecane, its solid phase prior to melting has been studied extensively [1,3,5–14], as noted above.

In this study we analyze the thermal expansivity α_p of solid hexadecane using the experimental data [1] and calculate the thermodynamic quantities such as the isothermal compressibility κ_T and the specific heat C_p near the melting point in solid hexadecane. By relating the specific heat C_p to the thermal expansivity α_p and α_p to κ_T, we examine the Pippard relations in solid hexadecane near the melting point.

In Section 2, we give our calculations and results. We discuss our results in Section 3. Finally, conclusions are given in Section 4.

The critical behaviour of the thermodynamic quantities such as the thermal expansivity α_p, isothermal compressibility κ_T and the specific heat C_p can be described by a power-law formula close to the melting point in solid hexadecane.

The thermal expansivity can be described close to the melting point by

$$\alpha_p = A(P - P_m)^{-\gamma} \tag{1}$$

where γ is the critical exponent for α_p, A is the amplitude and P_m is the melting pressure, as given previously [1]. Using the thermodynamic relation

$$\alpha_p/\kappa_T = (\partial P_m/\partial T)_V \tag{2}$$

the pressure dependence of the isothermal compressibility can be expressed as

$$\kappa_T = A(P - P_m)^{-\gamma}/(\partial P_m/\partial T)_V \tag{3}$$

by means of eq. (1).

Using the definition of the isothermal compressibility $\kappa_T = -(1/V)(\partial V/\partial P)_T$, the pressure

dependence of the volume of solid hexadecane will be obtained as follows :

$$V_T(P) = V_m \exp\left[1/(\partial P_m/\partial T)_V \left(A(P - P_m)^{1-\gamma}/1-\gamma\right)\right] \tag{4}$$

where V_m denotes the melting volume. Finally, using the thermodynamic relation

$$C_p = TV\alpha_p(\partial P_m/\partial T)_s \tag{5}$$

the pressure dependence of the specific heat can be obtained as

$$C_p = ATV(\partial P_m/\partial T)_s(P - P_m)^{-\gamma}. \tag{6}$$

Close to the melting point in solid hexadecane, the specific heat C_p can be related to the thermal expansivity α_p linearly, according to the first Pippard relation

$$C_p \doteq TV(dP_m/dT)\alpha_p + T(dS/dT)_m. \tag{7}$$

Similarly, the thermal expansivity α_p can be related linearly to the isothermal compressibility by the relation

$$\alpha_p = (dP_m/dT)\kappa_T + 1/V(dV/dT)_m \tag{8}$$

according to the second Pippard relation close to the melting point in solid hexadecane. Thus, linear plots of C_p against α_p (eq. 7) and α_p against κ_T (eq. 8) can be obtained near the melting point in solid hexadecane. By means of eqs. (7) and (8), the slope dP_m/dT can be deduced close to the melting point in this organic compound.

In order to establish the Pippard relations (eqs. 7 and 8), we first calculated the pressure dependence of the isothermal compressibility κ_T and of the specific heat C_p for solid hexadecane near the melting point.

We started by analyzing the experimental data for the thermal expansivity α_p at various pressures in solid hexadecane for constant temperatures of 302 and 325 K [1]. This analysis was performed according to the power-law formula (eq. 1) in a logarithmic form

$$\ln \alpha_p = \ln A - \gamma \ln(P - P_m). \tag{9}$$

Pruzan *et al* [1] have analyzed their experimental data for α_p. Here we reanalyzed their observed data and obtained the values of the critical exponent γ and the amplitude A for the solid phase of hexadecane, as tabulated in Table 1.

Table 1. Values of the critical exponenet γ for the thermal expansivity α_p and the amplitude A (eq. 1) and the values of the slope dP_m/dT (eq. 7) for the solid face of hexadecane close to the melting point for constant temperatures indicated.

T(K)	γ	$A \times 10^{-4}$ (MPa/K)	(dP_m/dT) (MPa/K)
302	0.54 ± 0.01	149.88	10.2 ± 0.2
325	0.53 ± 0.01	139.73	10.3 ± 0.2

From our analysis of the thermal expansivity α_p (eq. 9), we then calculated the pressure dependence of the isothermal compressibility κ_T using eq. (3) where we used the experimental value of $(\partial P/\partial T)_v - (\partial P/\partial T)_s$ = 10 MPa/K [1]. We also needed to evaluate the pressure dependence of the volume in solid hexadecane for the specific heat C_p. The volume values were evaluated as a function of pressure using eq. (4). In the $V_T(P)$ expression we used the experimental values of 1.305 cm^3/g at 30°C and 1.328 cm^3/g at 50°C [43] for the melting volume V_m. Those observed values for V_m corresponded to 294.930 cm^3/mole at 303 K and 300.128 cm^3/mole at 323 K for the solid hexadecane ($C_{16}H_{34}$) since it has 226 g in one mole. Thus the values of the volume $V_T(P)$ which we calculated as a function of pressure for constant temperatures of 302 and 325 K, were used in the C_p relation (eq. 6). We were then able to calculate the pressure dependence of the specific heat C_p in the same pressure range close to the melting point in solid hexadecane using eq. (6). Thus, we established the first Pippard relation (eq. 7) by plotting C_p against α_p for the solid phase of solid hexadecane for constant temperatures of T = 302 K and T = 325 K, as given in Figures 1 and 2 respectively. From our plots (Figures 1 and 2) we deduced the values of the slope dP_m/dT, as given in Table 1.

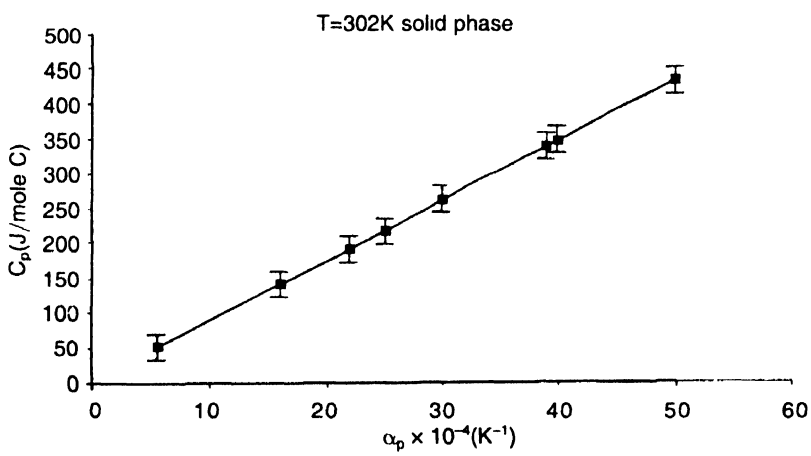

Figure 1. Specific heat C_p as a function of thermal expansivity α_p at various pressures close to the melting point for a constant temperature of T = 302 K in the solid phase of solid hexadecane (eq.7).Observed α_p values [1] are shown here.

We also established a linear variation of the thermal expansivity α_p with the isothermal compressibility κ_T according to the second Pippard relation (eq. 8) close to the melting point in solid hexadecane. Figures 3 and 4 give our α_p *vs.* κ_T plots for the solid phase of hexadecane at various pressures for constant temperatures of 302 and 325 K, respectively. Our values of the slope dP_m/dT, which we deduced from those plots (Figures 3 and 4) have the same value of 10 MPa/K, as measured experimentally [1].

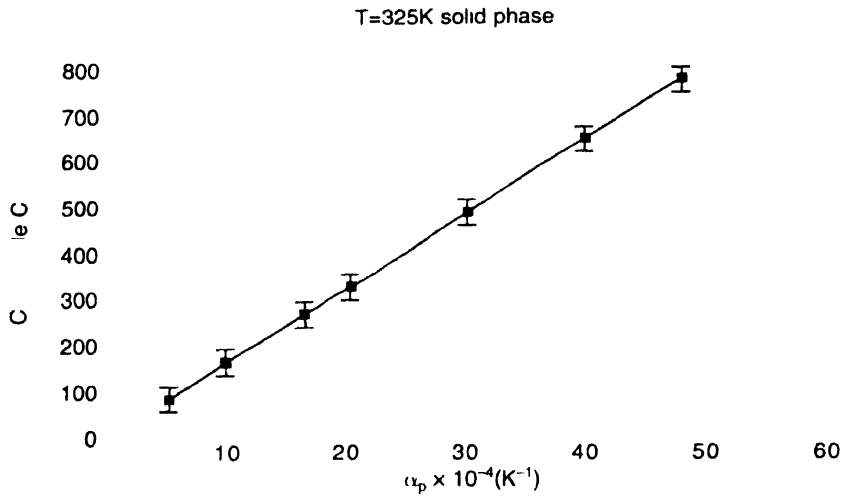

Figure 2. Specific heat C_p as a function of thermal expansivity α_p at various pressures close to the melting point for a constant temperature of T = 325 K in the solid phase of hexadecane (eq.7) *M.* Observed α_p values [1] are shown here.

We analyzed in this study the pressure dependence of the thermal expansivity α_p in the solid phase of hexadecane close to the melting point by a power-law formula (eq. 1). Using those values of the fitted parameters γ and A (Table 1), we first calculated the pressure dependence of the volume (eq 4) where we used observed values of the volume at 30 and 50°C as the melting volumes for solid hexadecane Those values of the volume were measured at nearly the same temperatures as 302 and 325 K respectively, at which the thermal expansivity α_p was measured [1]. Using those values of γ and A for the solid phase of hexadecane (Table 1), we were able to calculate the pressure dependence of the isothermal compressibility κ_T (eq. 3) and the specific heat C_p (eq. 6) near the melting point in solid hexadecane. When we plotted the specific heat C_p against the thermal expansivity α_p (eq. 7) and α_p against the isothermal compressibility κ_T (eq. 8), we obtained that they exhibit linear variations close to the melting point in this system. This indicates that α_p, κ_T and C_p show similar critical behaviour close to the melting point in solid hexadecane. When we extracted the values of the slope dP_m/dT from our plots, we obtained that they were almost the same as the observed value of 10 MPa/K. This also indicates that those thermodynamic quantities, namely, the thermal expansivity α_p, isothermal compressibility κ_T and the specific heat C_p exhibit similar critical behaviour described by the power-law formulas (eqs. 1, 3, 6) close to the melting point in solid hexadecane. Regarding the uncertainties in the measurements of the thermal expansivity α_p at various pressures for constant temperatures of 302 K and 325 K [1], we obtained the critical exponent within the uncertainties as $\gamma = 0.54 \pm 0.01$ and $\gamma = 0.53 \pm 0.01$, respectively (Table 1), from our analysis (eq. 1). When we calculated the isothermal compressibility κ_T (eq. 3), volume $V_T(P)$ (eq. 4) and the specific heat C_p (eq. 6) as a function of

pressure, we also obtained their values within the uncertainties. Those uncertainties are shown when C_p was plotted against α_p according to eq. (7) in Figures 1 and 2, and α_p was plotted against κ_T according to eq. (7) in Figures 3 and 4. From those plots, we extracted the values of the slope with the uncertainties given by dP_m/dT = 10.2 ± 0.2 and dP_m/dT = 10.3 ± 0.2 for constant temperatures of 302 K and 325 K, respectively, in the solid phase of hexadecane, as given in Table 1.

As we see from Table 1, we get the same value of about γ = 0.5 for the critical exponent from our analysis of the thermal expansivity α_p at the constant temperatures of 302 and 325 K in solid hexadecane. We analyzed in this study the experimental data for the thermal expansivity α_p at various pressures at the constant temperatures of 302 K and 325 K [1], as pointed out earlier. When the measurements are performed for the thermal expansivity α_p for other temperatures, they can also be analyzed according to a power-law formula (eq. 1) As examples, the experimental measurements were carried out for the thermal expansivity α_p of solid benzene at constant temperatures of 268, 302, 325 and 355 K, and of carbon tetrachloride at 283, 301.5, 302 and 325 K near the melting point, as functions of pressure [1]. They were analyzed according to the power-law and the values of the critical exponent γ for α_p were found as 0.3 and 0.15 for the solid phases of benzene and carbon tetrachloride, respectively for the constant temperatures indicated above. This means that the thermal expansivity α_p has the same pressure dependence with respect to the melting pressure p_m, independent of the constant temperatures, giving the same value as the universal critical exponent in benzene and carbon tetrachloride. On this basis, we expect that the universal exponent value of 0.5 should be obtained for other temperatures in solid hexadecane. This supports our conclusion that the solid hexadecane exhibits a second order phase transition with the exponent value of γ = 0.5 for the thermal expansivity α_p prior to melting. This value of γ = 0.5 from our analysis, is the same as the value of 1/2 for the critical exponent expected from theoretical models [44,45]. It has been obtained by Speedy [45] for superheated and supercooled water that the thermodynamic divergences of the thermal expansivity α_p, the specific heat C_p and the isothermal compressibility κ_T are described by

$$\alpha_p \sim C_p \sim \kappa_T \sim (|p - p_s(T)|)^{1/2} \text{ along an isotherm}$$

with the critical exponent γ = 0.5. The same exponent value was also obtained previously by Compagner [44] by analyzing the ferromagnetic spinodal in the mean field approximation and it was pointed out that the same exponent would be expected from a mean field theory of the gas and liquid spinodals. Similarly, the divergence behaviour of α_p, C_p and κ_T for the solid phase of hexadecane studied here, which was described according to the power-law formula (eq. 1), would give the same exponent value of γ = 0.5, as obtained from the model studies [44,45]. Our analysis using the experimental data for the thermal expansivity α_p of solid hexadecane gives the values of 0.54 (302 K) and 0.53 (325 K), which agree with the mean field value of 0.5. As

indicated above, this exponent value can be considered as a universal value which is independent of the temperature for the solid hexadecane under the pressure conditions given here Also, this exponent value of 1/2 is expected from a power-law formula for the thermal expansivity in NaCl using a model based on quasiharmonic theory close to melting in alkali halides [46,47], as also pointed out previously [1].

Since the isothermal compressibility κ_T and the specific heat C_p were calculated using our γ values (Table 1) by means of eqs (3) and (6), respectively, as indicated above, the Pippard relations (eqs 7 and 8) when plotted in Figures (1–4) have zero intercept values In order to have nonzero values of the intercept $T(dS/dT)_m$ (eq 7)

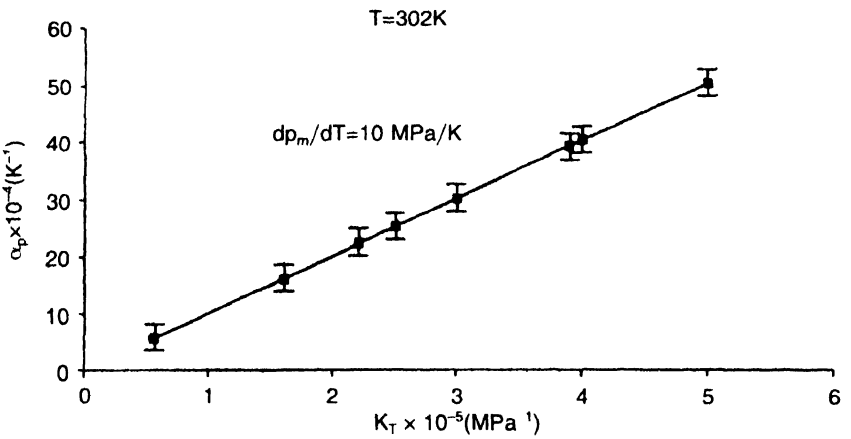

Figure 3. Thermal expansivity α_p as a function of the isothermal compressibility κ_T close to the melting point in the solid phase of hexadecane at various pressures for a constant temperature of T = 302 K (eq 8) Observed α_p values [1] are shown here

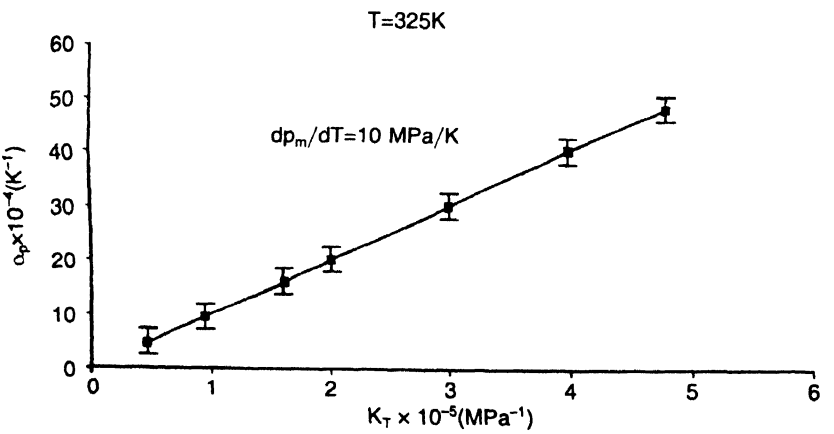

Figure 4. Thermal expansivity α_p as a function of the isothermal compressibility κ_T close to the melting point in the solid phase of hexadecane at various pressures for a constant temperature of T = 325 K (eq 8) Observed α_p values [1] are shown here

and $(dV/dT)_m/V$ (eq. 8), we should know the temperature dependence of the entropy S and of the volume V, at the melting point in solid hexadecane. This will shift the plots of C_p against α_p (Figures 1 and 2) and of α_p against κ_T (Figures 3 and 4), but the slope dP_m/dT will remain the same.

When the volume is measured or calculated as a function of temperature in the solid phase of hexadecane, then the Pippard relations (eqs. 7 and 8) can also be established at various temperatures near the melting point for constant pressures in this system.

Those thermodynamic relations studied here, which are essentially derived for λ-phase transitions [41,42], can be established in the case of divergence of α_p, κ_T and C_p near the melting point in solid hexadecane. If this molecular organic compound contains defects and impurities, the melting temperature (or melting pressure) will then be determined in an interval, which can be defined as a heterophase melting [3] where continuous large variations occur in the thermodynamic quantities [1]. Thus, experimentally, a pure solid hexadecane is required to shorten the melting zone [4] where the thermodynamic quantities can be measured as they diverge, which can then be analyzed according to a power-law formula. Also, measurements in the melting zone of solid hexadecane, as in other molecular organic compounds, should require a long time to reach equilibrium [1].

We have calculated here the pressure dependence of the volume, $V_T(P)$, the isothermal compressibility κ_T and the specific heat C_p for solid hexadecane close to its melting point. This calculation was carried out by analyzing the thermal expansivity measured as a function of pressure for constant temperatures of 302 and 325 K in this organic compound.

We have obtained here a linear variation of the specific heat C_p with the thermal expansivity α_p (first Pippard relation) and also a linear variation of α_p with the isothermal compressibility κ_T (second Pippard relation) near the melting point for solid hexadecane. Our values of dP_m/dT extracted from those linear plots, agree well with the observed value for this system.

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